## (54) ANTI-ELECTROSTATIC THERMOPLASTIC MOULDING COMPOSITIONS AND MOULDINGS

(71) We, CHEMISCHE WERKE HÜLS AKTIENGESELLSCHAFT, a German Company, of 4370 Marl, Germany, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following Statement: —

The invention relates to anti-electrostatic moulding compositions and mouldings of polyolefins which contain fatty acid glycerides and glycerol.

It is known to incorporate polyhydric alcohols and fatty acid glycerides, simultaneously with 3 to 20 per cent by weight of an adsorbent in the form of a pigment or filler, into thermoplastic materials such as polyethylene (U.S. Patent Specification 2,758,984). It is also known from German Published Specification 1,271,391 to mix polyolefins with monoglycerides and diglycerides containing less than 1.5% of glycerol, to prevent haze formation in films. Finally, it is known to use fatty alkyldialkanolamines for the antistatic finishing of polyolefins, and in particular to use the former either alone (U.S. Patent Specification 2,992,199) or in combination with further antistatically active compounds (German Published Specification 1,230,210 and German Published Specification 1,234,020).

The use of polyhydric alcohols, such as for example glycerol, is completely unsatisfactory from the point of view of an antistatic effect, as will be shown later. On the other hand, glycerol esters show greatly differing behaviour.

German Published Specification 1,927,677 discloses that the monoglycerides of saturated $C_{12}$ to $C_{18}$ fatty acids can be used as anti-static agents, but do not have a conspicuously good effect. In order to obtain particularly effective monoglycerides, this German Published Specification proposes the use of $C_6$ to $C_{10}$ fatty acids and demonstrates that such products should be as little contaminated by glycerol as possible. For this reason, a process for the manufacture of 100% pure monoglycerides is also indicated.

Fatty alkylalkanolamines remain practically completely ineffective at concentrations below 0.2 per cent by weight. Only in the case of high pressure polyethylene is a slight activity detectable. At higher doses, namely, $\geq 1\%$, they cause, on the other hand, sticky, oily-smeary coatings on the surfaces of the mouldings. Combinations with other antistatically active compounds hence represent a real necessity for these substances.

For various reasons, it is desirable that particularly the crude reaction products from glycerol on the one hand and longer fatty acids or their natural triglycerides on the other hand, which products in part contain a great deal of glycerol, should be rendered utilisable, though, according to the state of the art, there exists a prejudice against the use of glycerol-rich mixtures and against the use of longer-chain fatty acid glycerides.

According to the present invention there are provided antistatic moulding compositions and mouldings of polyolefins, containing a mixture of

(a) 60 to 96 per cent by weight of a glycerol monoester of a fatty acid with 12 to 26 carbon atoms, in which the content of diester and triester is at most 25 per cent by weight of the glycerol-glycerol ester mixture, and

(b) 40 to 4 per cent by weight of glycerol.

According to a particular embodiment of

the invention, the mouldings and moulding compositions can additionally contain (c) an amine of the formula

$$R—(OCH_2—CHOH—CH_2)_z—N \begin{array}{c} R_1 \\ \diagup \\ \diagdown \\ R_2 \end{array}$$
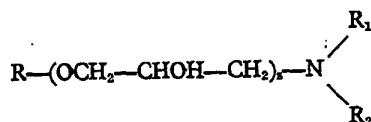
5 wherein R denotes an alkyl or alkenyl radical with 6 to 26 carbon atoms, $R_1$ and $R_2$ denote the radical $(C_xH_{2x}O)_n$ with $x=2$ or 3 and $n=$ an integer from 1 to 7, and $R_2$ can alternatively denote hydrogen, and $z=0$, or 1.

10 The mixtures of compounds (a) and (b) and optionally (c) can, on the one hand, be applied to the surfaces of the mouldings or moulding compositions (for example by dipping or spraying the mixtures, dissolved in water or

15 organic solvents if desired). On the other hand, they can be incorporated into the moulding compositions suitably in an amount of 0.2 to 2.8 per cent by weight, relative to the moulding compositions.

20 Suitable polyolefins which can be provided with an anti-electrostatic finish by the additives are, for example, high pressure and low pressure polymers of ethylene, propylene, 1-butene and 1-pentene, especially all types of

25 polythylene with $\eta_{red}$ values of about 0.4 to 4.0, corresponding to average molecular weights of about 20,000 to 200,000, polypropylenes with $\eta_{red}$ values of about 1.5 to 7.0, corresponding to average molecular weights

30 of about 150,000 to 700,000, and poly-1-butenes with $\eta_{red}$ values of about 1.0 to 6.0, corresponding to average molecular weights of about 500,000 to 3,000,000, which are manufactured according to known process

35 techniques (see R. Vierweg and A Müller, Kunststoffe-Handbuch (Plastics Handbook), volume IV, Karl-Hanser-Verlag, Munich 1966), as well as any desired copolymers and polymer mixtures thereof.

40 The total content of the anti-electrostatic mixture in the moulding compositions and mouldings is desirably 0.2 to 2.8 per cent by weight, preferably 0.4 to 2.0 per cent by weight.

45 As component (a) of the mixture of (a) and (b), which is employed alone or as a further mixture with the component (c), the monoesters of glycerol and fatty acids having 12 to 26 carbon atoms, especially 12 to 18 carbon

50 atoms, and above all 14 carbon atoms, are used. These monoesters can still contain minor proportions of diesters and/or triesters, for example remaining from their manufacture. These proportions should not exceed

55 25 per cent by weght, relative to the mixture of (a) and (b). The component (a) is employed in an amount, relative to the total amount of anti-electrostatically active mixture, of 60 to

60 96 per cent by weight, preferably 70 to 90 per cent by weight.

The glycerol (component (b)) is employed in an amount, relative to the total amount of anti-electrostatically active mixture, of 40 to 4.0 per cent by weight, preferably 30 to

65 10.0 per cent by weight.

As component (c), one or more amines of the formula

$$R—(OCH_2—CHOH—CH_2)_zN \begin{array}{c} R_1 \\ \diagup \\ \diagdown \\ R_2 \end{array}$$
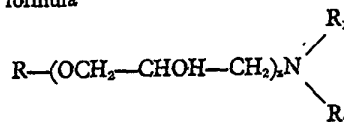
are employed, wherein R denotes an alkyl or

70 alkenyl radical with 6 to 26 carbon atoms, preferably 10 to 18 carbon atoms, especially 10 to 14 carbon atoms; $R_1$ and $R_2$ denote the grouping $(C_xH_{2x}O)_nH$ with $x=2$ or 3, preferably 2, $n=$ an integer from 1 to 7, preferably

75 1 or 2, and $R_2$ can alternatively denote hydrogen, and $z=0$ or 1.

This component (c) is advantageously employed in amounts of 0.05 to 0.8, per cent by weight, preferably 0.1 to 0.5 per cent by

80 weight, within the total amount of anti-electrostatic additive (0.2 to 2.8 per cent by weight), based on the moulding or moulding composition.

The antistatically active glycerol-glycerol

85 ester combination may be obtained either by glycerolysis of fats and oils, or by starting from the reagents, glycerol and fatty acid, which are in an anhydrous form. For example, the fatty acid may be heated with an about

90 7-fold to 8-fold molar excess of glycerol to about 200 to 220°C (Hundeshagen, J. pr. (2) 28, 225) or the Na salts or K salts of such fatty acids may be reacted with an α- or β-halogenopropylene glycol (see, for example,

95 Guthe, Z.B. 44, 83), and the requisite amount of glycerol is in each case added finally. The following may be mentioned as usable fatty acid components. arachidic acid, eicosanecarboxylic acid, behenic acid, lignoceric acid

100 and especially lauric acid, myristic acid, palmitic acid, oleic acid and stearic acid, whilst as glycerides containing appropriate amounts of glycerol, α- or β- or (α+β)-glycerides may be mentioned, namely β-monoarachidin, β-

105 monobehenin, α-lignocerin, especially α-monostearin, β-monostearin, (α+β)-monostearin, α-monopalmitin, (α+β)-monopalmitin, α-monomyristin, α-monoolein, (α+β)-monoolein, and mixtures of these, such as (α or β

110 or α+β)-monostearin/monopalmitin or (α or β or α+β)-monostearin/monoolein or (α or β or α+β or α+β)-monopalmitin/monostearin/monoolein or (α or β or α+β)-monolaurin/monomyristin.

115 In the case of $z=0$, suitable amines (component (c)) may be obtained by oxyethylation or oxypropylation of appropriate amines which

have for example been obtained either from an alcohol via the halogenalkane or halogenalkene, or been synthetised via a Hofmann acid amide degradation, whilst in the cast of $z=1$,
5  suitable amines may be obtained by addition of appropriately oxyethylated or oxypropylated primary or secondary amines to glycidyl ethers of the general formula

$$R—OCH_2—CH—CH_2$$
$$\diagdown O \diagup$$
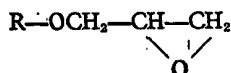
10  in which R is as defined above.

It does not require special underlining that in principle it is also possible for several representatives of this class of substance to be employed simultaneously. Suitable amines are,
15  for example, the monoethylene oxide adduct of hexacosylamine, the di-(-ethylene oxide) adduct of nonadecyl - N - ($\beta$ - hydroxyethyl)-amine, the tetra-(ethylene oxide) adduct of hexadecylamine, the hepta(ethylene oxide)
20  adduct of decylamine, the penta(propylene oxide) adduct of undecen - (10) - yl - amine, N - (3 - decyloxy - 2 - hydroxypropyl) - N-ethanolamine, N - (3 - nonadecyloxy) - 2-hydroxypropyl) - N - diethanolamine and
25  especially dodecyl-diethanolamine, tridecyl-diethanolamine, pentadecylmonoethanolamine and pentadecyldiethanolamine, heptadecyl-monoethanolamine and heptadecyl-diethanol-amine, octadecyldiethanolamine, N - (3-
30  dodecyloxy - 2 - hydroxypropyl) - mono-ethanolamine, N - (3 - dodecyloxy - 2-hydroxy - 2 - hydroxypropyl) diethanolamine and the bis-hepta(ethylene oxide) adduct of hexylamine.
35  The antistatically active substances are appropriately incorporated into the polyolefin which is in a pulverulent form, for example with the aid of a simple mixer. The resulting mixture may then be charged into the pro-
40  cessing machines either directly or after prior densification into granules, and shaped in the machines within the temperature range of 120 to 300°C applicable to polyolefins. In order to achieve good and rapid distribution, it is some-
45  times advantageous to introduce the antistatically active compounds in a dissolved form. It is also possible to incorporate the antistatic agent into the polyolefin directly on a mill or, for example, in an extruder in the case of
50  injection moulding.

Another method which has proved successful is firstly to manufacture granules of high concentration of antistatic agent and to bring these to the desired content of antistatic agent
55  on processing by admixing granules which are free of additives.

It is possible to add further additives which are otherwise customary in plastics processing, for example dyestuffs, stabilisers, plasticisers,
60  extenders and fillers as well as lubricants.

Customary stabilisers which may be mentioned are phenolic antioxidants, such as for example 2,2' - thio - bis - (4 - methyl - 6-tert . butyl - 5 - octylphenol) (0.1 to 0.2 per cent by weight), optionally together with sul-  65 phur-containing compounds, such as dilauryl thiodipropionate.

It can also be advisable to add to the poly-olefin additional substances which prevent a slight yellowing of the amine component on  70 prolonged exposure to high temperatures. Suitable stabilisers of this nature are, for example, esters of phosphorous acid, especially didecylphenyl phosphite, decyldiphenyl phosphite, triphenyl phosphite, tris-(nonyl-  75 phenyl phosphite, and tris((nonylphenol+9 mols of ethylene oxide) phosphite, which are added in amounts of 0.01 to 0.4 per cent by weight relative to the polyolefin. An addition of alkanesulphonates, for example sodium  80 pentadecanesulphonate, also has a flavourable effect in the same way. Approx. 0.01 to 0.4 per cent by weight, relative to the polyeolefin, are required for this purpose.

These additives as a rule do not influence  85 the antistatic effect. Should such nevertheless sometimes be the case as a result of a very high proportion of filler consisting of chalk, glass fibres or asbestos a somewhat higher proportion of the anti-electrostatically active  90 additive easily and rapidly provides a remedy.

The anti-electrostatic behaviour of the mouldings is ascertained, inter alia, by means of a simple manual test which can be employed quite generally for assessing the anti-  95 electrostatic behaviour of goods which have been provided with a finish, and which is referred to briefly as the ash test. The result of this ash test is regarded as positive (sign "+" — it thus relates to the effect of the  100 incorporated substance), if, at 50% relative humidity and 23°C, the vigorously rubbed article no longer attracts ash particles at a distance of 1 cm from the support on which the ash rests. Further, the sign — denotes  105 that ash is attracted to a great extent, and (—) that ash is still attracted.

In this test, observation of the atmospheric humidity is above all of importance for an objective assessment, since the humidity in-  110 fluences the charging behaviour to a very particularly marked extent.

It is surprising here that mixtures of (a) and (b) give an excellent and reproducible anti-electrostatic effect within the range  115 claimed. What is above all surprising is however that glycerol, which when used by itself is completely ineffective, represents the decisive parameter in this synergism. It is also valuable that, as a result of an additional  120 synergistic effect, the amines (component (c)) make it possible to reduce the concentration relative to the amine-free glycerol-glycerol ester mixture.

The Tables which follow make clear what has been said about the synergism; in particular, Table I firstly compares rectangular dish mouldings of partly crystalline propylene homopolymer and copolymer treated in different ways (Examples 2 to 20), both with one another and also with the base material which has not been provided with a finish (O-samples in Example 1). Table II (poly-1-butene) and Table III (polyethylene) are compiled similarly. In each case, the O-samples are given first, with data regarding the density and the $\eta_{red}$ value of the polymer used, with the reduced specific viscosity relative to c = 0.1 at 110°C in p-xylene.

Apart from the ash test, the surface resistance (measured according to DIN 5343) and the decay, followed by means of a measuring head according to Schwenkhagen (see Textil-praxis 12/11, page 1147 (1957)), of the charge produced on the test specimens suspended in a clamping device by rubbing them vigorously with a cotton cloth, represent the most important criteria.

Firstly, Table I shows that the high surface resistance and hence the charging behaviour of the starting material (Example No. 1) cannot be favourably influenced by adding glycerol (Example 2). Whilst the use of glycerol monoesters is able to reduce the surface resistance, it can only do so to a very unsatisfactory extent (Examples 3, 4 and 6). Equally unsatisfactory results are also obtained if lauryldiethanolamine or N - (3 - dodecyloxy-2 - hydroxypropyl) - ethanolamine is employed in a concentration according to Example 13, by itself or combined with glycerol or glycerol monoesters (Examples 14 and 15), though here admittedly the somewhat more favourable behaviour of the combination with mono-esters (Example 15) as against that with glycerol (Example 14) again becomes noticeable, analogously to Example 2 and 4.

Highly surprising findings are undoubtedly those of Examples 16 to 18. These quite unmistakably concern synergistic effects which —as is shown by Example 18— are not unfavourably influenced even by the presence of amounts of diesters and triesters which are to be regarded as an impurity from the manufacturing process and which by themselves are again completely inactive, analogously to glycerol, this absence of an unfavourable influence applying as long as the proportion of diesters and triesters in the glycerol-glycerol ester mixture does not exceed 25, and prefer-

ably dos not exceed 20, per cent by weight (comparison Example 20). If the glycerol content exceeds 40 per cent by weight, relative to the glycerol-glycerol ester mixture (Example 7 and 19), the total effect of the combination is lost. For optimum mixtures, the glycerine content should not exceed 25 per cent by weight and should not be less than 10 per cent by weight, of the glycerol-glycerol ester mixture.

If, in particular, it is less than 4.0 per cent by weight, an increased activity relative to Examples, 3, 4 and 6 is no longer detectable.

Table I further shows that in a comparison of the acyl-radicals in the monoglycerides with one another, those with 12, and especially 14, carbon atoms prove best (Examples 8 and 11). Higher fatty acid radicals can also still be used (Examples 9 and 10). Compounds with fatty acid radicals which are too short are of little use if glycerol is used conjointly (Example 12).

Table II summarises the results in relation to injection mouldings of poly-1-butene. It is conspicuous that in comparison to poly-propylene, somewhat higher doses are required here to obtain results agreeing approximately with Table I. Again Example 4 and — in agreement therewith — Examples 7 and 8 show a distinct synergism which in the light of the findings of Examples 2 and 3, as well as 5 and 6, represents a real surprise. Comparison Example 9 shows that the presence of major amounts of glycerol, analogously to Examples 7 and 19 of Table I, has an unfavourable effect.

Table III characterises the behaviour of blow-mouldings and blown films, of a low pressure polyethylene of the nature specified in Example 1, which have been treated in various ways. It is seen that a glycerol-glycerol ester mixture according to Example 4, consisting of 85% of glycerol monolaurate and 15% of glycerol, is distinctly superior to the effect of the individual components (compare to Example 2 and 3), and that combinations with amines of the nature indicated (Example 5 and 6) allow the concentration of the additives to be reduced further. In the case of the manufacture of flat films (Example 7) a total concentration of 0.7 part per 100 parts of the polymer already suffices to reduce the surface resistance to $10^9\Omega$ and hence to achieve a satisfactory antistatic behaviour.

The terms and signs used in the tables denote:

| x) | very slow: | half-life $>120''$ |
| | slow: | half-life $> 60''$, $<120''$ |
| | rapid: | half-life $> 10''$, $< 60''$ |
| | very rapid: | half-life $> 10''$ |
| xx) | — | = ash is strongly attracted |
| | (—) | = ash is still attracted |
| | + | = no ash is attracted |

TABLE I

Injection Mouldings of Polypropylene

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in $\Omega$ at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay*) | Ash test**) at 50% relative atmospheric humidity and 23°C. | Notes |
|---|---|---|---|---|---|---|
| 1. | 0 | Polypropylene: (injection moulding grades) a) propylene homopolymer (D = 0.906, $\eta$ red = 4) or b) propylene–ethylene copolymer with 5 per cent by weight of ethylene (D = 0.903, $\eta_{red}$ = 4) | $>10^{18}$ | very slow | — | O-sample |
| 2. | 1.0 | Glycerol | $>10^{18}$ | very slow | — | comparison example |
| 3. | 1.0 | Glycerol-($\alpha$)-monomyristate | $4 \cdot 10^{11}$ | slow | (—) | comparison example |
| 4. | 1.0 | Glycerol-($\alpha$)-monostearate | $3 \cdot 10^{12}$ | slow | (—) | comparison example |
| 5. | 1.0 | Glycerol-($\alpha$)-monocaprylate | $3 \cdot 10^{11}$ | slow | (—) | comparison example |
| 6. | 1.0 | Glycerol-($\alpha$)-monolaurate | $6 \cdot 10^{11}$) | slow | (—) | comparison example |

TABLE I (Continued)

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in Ω at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay*) | Ash test**) at 50% relative atmospheric humidity and 23°C (−) | Notes |
|---|---|---|---|---|---|---|
| 7. | 0.5 | Glycerol-(α)-monostearate | $7 \cdot 10^{12}$ | slow | (−) | comparison example |
| 8. | 0.5 | Glycerol | $4 \cdot 10^{9}$ | very rapid | + | is claimed |
| 9. | 0.75 / 0.25 | Glycerol-(α)-monomyristate / Glycerol | $2 \cdot 10^{10}$ | rapid | + | is claimed |
| 10. | 0.75 / 0.25 | Glycerol-(α)-monostearate / Glycerol | $7 \cdot 10^{10}$ | relatively rapid | + | is claimed |
| 11. | 0.75 / 0.25 | Glycerol-(α)-monobehenate / Glycerol | $6 \cdot 10^{9}$ | very rapid | + | is claimed |
| 12. | 0.75 / 0.25 | Glycerol-(α)-monolaurate / Glycerol / Glycerol-(α)-monocaprylate / Glycerol | $3 \cdot 10^{12}$ | slow | (−) | comparison example |
| 13. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $>10^{13}$ | very slow | − | comparison example |

TABLE I (Continued)

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in Ω at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay*) | Ash test**) at 50% relative atmospheric humidity and 23°C. | Notes |
|---|---|---|---|---|---|---|
| 14. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $>10^{13}$ | very slow | — | comparison example |
|  | 0.5 | Glycerol |  |  |  |  |
| 15. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $5 \cdot 10^{11}$ | slow | (—) | comparison example |
|  | 0.5 | Glycerol-(α)-monostearate |  |  |  |  |
| 16. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $2 \cdot 10^{9}$ | very rapid | + | is claimed |
|  | 0.5 | Glycerol-glycerol ester mixture of 80% of α-mono-stearate and 20% of glycerol |  |  |  |  |
| 17. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $6 \cdot 10^{8}$ | very rapid | + | is claimed |
|  | 0.5 | Glycerol-glycerol ester mixture of 80% of α-mono-laurate and 20% of glycerol |  |  |  |  |

TABLE I (Continued)

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in Ω at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay* | Ash test** at 50% relative atmospheric humidity and 23°C | Notes |
|---|---|---|---|---|---|---|
| 18. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $3 . 10^9$ | very rapid | + | is claimed |
|  | 0.5 | Glycerol-glycerol ester mixture, consisting of 65% of (α + β)-monostearate, 15% of distearate, 1% of tristearate and 19% of glycerol |  |  |  |  |
| 19. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $1 . 10^{12}$ | slow | (−) | comparison example |
|  | 0.5 | Glycerol-glycerol ester mixture, consisting of 55% of α-monostearate and 45% of glycerol |  |  |  |  |
| 20. | 0.1 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $4 . 10^{11}$ | slow | (−) | comparison example |
|  | 0.5 | Glycerol-glycerol ester mixture of 52% of (α + β)-monostearate, 34% of distearate, 3% of tristearate and 11% of glycerol |  |  |  |  |

TABLE II

Injection Mouldings of Poly-1-butene

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in $\Omega$ at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay*) | Ash test**) at 50% relative atmospheric humidity and 23°C. | Notes |
|---|---|---|---|---|---|---|
| 1. | 0 | Poly-1-butene (D= 0.915, $\eta$ red = 4) | $>10^{13}$ | very slow | — | O-sample |
| 2. | 1.5 | Glycerol | $>10^{13}$ | very slow | — | comparison example |
| 3. | 1.5 | Glycerol-($\alpha$)-monostearate | $5 \cdot 10^{12}$ | slow | (—) | comparison example |
| 4. | 1.5 | Glycerol-glycerol ester mixture consisting of: 65% of monostearate, 15% of distearate, 1% of tristearate and 19% of glycerol | $5 \cdot 10^{9}$ | rapid | + | is claimed |
| 5. | 0.2 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $>10^{13}$ | very slow | — | comparison example |
| | 0.7 | Glycerol | | | | |

TABLE II (Continued)

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in $\Omega$ at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay*) | Ash test**) at 50% relative atmospheric humidity and 23°C. | Notes |
|---|---|---|---|---|---|---|
| 6. | 0.2 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $5 \cdot 10^{12}$ | slow | (—) | comparison example |
| | 0.7 | Glycerol-($\alpha$)-monostearate | | | | |
| 7. | 0.2 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $10^9$ | very rapid | + | is claimed |
| | 0.7 | Glycerol-glycerol ester mixture consisting of 80% of $\alpha$-monostearate and 20% of glycerol | | | | |
| 8. | 0.2 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $10^9$ | very rapid | + | is claimed |
| | 0.7 | Glycerol-glycerol ester mixture consisting of: 65% of monostearate, 15% of distearate, 1% of tristearate and 19% of glycerol | | | | |

TABLE II (Continued)

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in Ω at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay*) | Ash test**) at 50% relative atmospheric humidity and 23°C | Notes |
|---|---|---|---|---|---|---|
| 9. | 0.2 | Dodecyldiethanolamine or N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $5 . 10^{13}$ | slow | (−) | comparison example |
|  | 0.7 | Glycerol-glycerol ester mixture consisting of 55% of α-monostearate and 45% of glycerol |  |  |  |  |

TABLE III

Blow Mouldings and Films of Polyethylene
(Examples 2 to 6 relate to Blow Mouldings and Blown Films; Example 7 — Flat Films)

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in Ω at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay*) | Ash test**) at 50% relative atmospheric humidity and 23°C | Notes |
|---|---|---|---|---|---|---|
| 1. | 0 | Low pressure polyethylene (D = 0.948, η_red = 2.4) | $>10^{13}$ | very slow | − | O-sample |
| 2. | 1.5 | Glycerol-(α)-monolaurate | $10^{13}$ | slow | (−) | comparison example |

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TABLE III (Continued)

| Example No. | Parts per 100 parts of polymer | Formulation | Surface resistance in Ω at 50% relative atmospheric humidity and 23°C, and 1 day after manufacture | Charge decay* | Ash test** at 50% relative atmospheric humidity and 23°C | Notes |
|---|---|---|---|---|---|---|
| 3. | 1.5 | Glycerol | $>10^{13}$ | very slow | – | comparison example |
| 4. | 1.5 | Glycerol-glycerol ester mixture consisting of 85% of α-monolaurate and 15% of glycerol | $3 \cdot 10^9$ | rapid | + | is claimed |
| 5. | 0.15 | Dodecyldiethanolamine | $1 \cdot 10^9$ | very rapid | + | is claimed |
| | 1.2 | Glycerol-glycerol ester mixture according to Example 4 | | | | |
| 6. | 0.15 | N-(3-dodecyloxy-2-hydroxy-propyl)-ethanolamine | $2 \cdot 10^9$ | very rapid | + | is claimed |
| | 1.2 | Glycerol-glycerol ester mixture according to Example 18 in Table I | | | | |
| 7. | 0.2 | Dodecyldiethanolamine | $2 \cdot 10^9$ | very rapid | + | is claimed |
| | 0.5 | Glycerol-glycerol ester mixture according to Example 4 | | | | |

WHAT WE CLAIM IS:—

1. Anti-electrostatic moulding compositions and mouldings of polyolefins, containing a mixture of

(a) 60 to 96 per cent by weight of a glycerol monoester of a fatty acid with 12 to 26 carbon atoms, which contains at most 25 per cent by weight of diester and triester relative to the glycerol-glycerol ester mixture, and

(b) 40 to 4 per cent by weight of glycerol.

2. Anti-electrostatic moulding compositions and mouldings according to Claim 1, additionally containing

(c) an amine of the formula
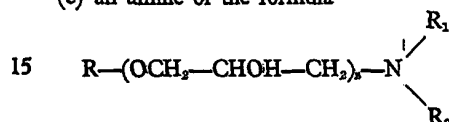
$$R-(OCH_2-CHOH-CH_2)_z-N{\Large\overset{\textstyle R_1}{\underset{\textstyle R_2}{\diagdown}}}$$

wherein R denotes an alkyl or alkenyl radical with 6 to 26 carbon atoms, $R_1$ and $R_2$ denote the radical $(C_xH_{2x}O)_nH$ where $x$ denotes 2 or 3 and n denotes an integer from 1 to 7, $R_2$ may also denote hydrogen, and $z$ denotes 0 or 1.

3. Anti-electrostatic moulding compositions and mouldings according to Claim 1 or 2, containing 0.2 to 2.8 per cent by weight, relative to the mouldings or moulding compositions, of the mixture of (a) and (b) and optionally (c).

4. Anti-electrostatic moulding compositions and mouldings according to Claim 3 containing 0.05 to 0.8 per cent by weight of (c).

5. A method of improving the anti-electrostatic behaviour of a polyolefin moulding or moulding composition which comprises applying to the surface of the moulding or moulding composition the mixture of (a) and (b) and optionally (c) as defined in claims 1 and 2.

6. A method of improving the anti-electrostatic behaviour of a polyolefin moulding or moulding composition which comprises incorporating into the moulding composition the mixture of (a) and (b) and optionally (c) as defined in claims 1 and 2.

7. Anti-electrostatic moulding compositions and mouldings of polyolefins substantially as described with reference to any of the foregoing Examples 8—11 and 16—18 of Table I, Examples 4, 7 and 8 of Table II and Examples 4—7 of Table III.

J. Y. & G. W. JOHNSON,
Furnival House,
14—18, High Holborn,
London, WC1V 6DE.
Chartered Patent Agents,
Agents for the Applicants.